

U.S. Department of Energy



Environmental Management Consolidated Audit Program

Module 4

Checklist for Radiochemistry

Audit ID: _____

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4.1	Standard Operating Procedures		
4.1.1	<p>Methods employed by the laboratory meet the following general guidelines:</p> <ul style="list-style-type: none"> are capable of producing data that meets the minimum method QA/QC requirements; and, can be referenced to nationally accepted sources such as EPA methods, DOE Methods Compendium, HASL 300 methods, etc.; and, are tested and validated with control samples and blanks prior to analysis. <p><i>(ISO 17025 5.4.2; NELAC 5.10.1, 5.10.2)</i></p>		
4.1.2	<p>SOPs are available at the laboratory workstations.</p> <p><i>(DOE Order 414.1 A, Criterion 4 and 5)</i></p>		
4.1.3	<p>Deviations from the SOPs are documented through a variance process monitored by the laboratory's Quality Assurance Manager.</p> <p><i>(ISO 17025 4.3.3)</i></p>		

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4.1.4	<p>All observations and results recorded by the laboratory are on pre-printed forms, electronic media, or entered into permanent laboratory logbooks.</p> <p><i>(ANSI N42.23, 5.25; ISO 17025 4.12.2.1; NELAC 5.11.3)</i></p>		
4.1.5	<p>At a minimum the SOPs define, establish and implement the following:</p> <ul style="list-style-type: none"> • matrices and limitations • hazards and safety (radiation, equipment and reagents); • instrumentation, equipment and materials; • solution (reagent make-up); • standardization and calibration (tracer and control solutions); • assessment of blanks; • assessment of controls; • assessment of duplicates; • MDA assessment; • sample pre-treatment and separations; • resin disposal; • glassware cleaning • calculations; • reporting results; • water purity; • SQA documentation; • emergency requirements; • disposal of material; and, 		

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	<ul style="list-style-type: none"> chemical storage. <p><i>(DOE Order 414.1A, Criterion 5; ISO 17025 5.4.4; NELAC 5.10)</i></p>		
4.1.6	<p>Laboratory SOP contains formula for calculating total propagated uncertainty including both systematic and random error.</p> <p><i>(ANSI N42.23, 4.1.3.2, and A8; ISO 17025 5.4.6)</i></p>		
4.1.7	<p>Laboratory SOP specifies acceptance criteria for QC samples. At a minimum the acceptance criteria is as follows:</p> <ul style="list-style-type: none"> batch blank: MDA is less than RDL unless all samples in the batch are positive; relative bias -.25 to +.25; duplicates: normalized absolute difference between sample and duplicate is ≤ 3; and, matrix spikes: Recoveries of 60 - 140%. <p><i>(ICPT BOA Radiochemistry Requirements Part 1 Sec. 2.3.1.4, 2.3.2.5, 2.3.3.3, and 2.3.4.4)</i></p>		

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4.1.8	Laboratory SOP defines corrective action requirements for QC samples that do not meet Acceptance Criteria. <i>(DOE Order 414.1A, Criterion 3; ANSI N42.23, 5.2.4, 5.2.9; ISO 17025 4.9; NELAC 5.10.1.2)</i>		
4.1.9	Analytical worksheets include, at a minimum: <ul style="list-style-type: none"> • name or initials of the person performing the analysis; • instrument used in the analysis (If the subcontract laboratory has more than one instrument of a particular model, a unique designation shall be given to each); • name or initials of the peer, supervisory, or QA reviewer; • calibration information for all analytical work (Radiochemistry counting instrument calibration information should be limited to calibration dates, computer data file names, and a statement certifying that calibrations were successfully performed on schedule); • information on standards used during the analysis; • analytical procedure used; • equations for calculations used to obtain results; and • date and time that the analysis was performed. <i>(NELAC 5.12.3.3)</i>		

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4.2	Training		
4.2.1	Laboratory personnel demonstrate adequate understanding of radiological controls and radioactive material handling procedures, emergency procedures and use of instrumentation. <i>(10 CFR 19.12, 20.1101 a)</i>		
4.3	Internal Tracers		
4.3.1	All samples (including QC samples), are spiked with a tracer, when applicable, that chemically mimics and does not interfere with the target analyte through radiochemical separations. <i>(ANSI N 42.23, 5.2.7; NELAC D4.1c)</i>		
4.3.2	Tracers or carriers are added at the very beginning of the sample preparation process to limit losses of analytes of interest early in the preparation process. For solid samples, the tracer is added after grinding, sieving, etc., but prior to any muffling or dissolution. <i>(ANSI N42.23, 5.27; NELAC D4.1c)</i>		

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4.3.3	<p>The identity and amount of tracer or carrier added to the sample is recorded.</p> <p><i>(NELAC D.4.1)</i></p>		
4.3.4	<p>The laboratory has established minimum acceptable tracer and carrier recoveries which meet the following requirements:</p> <ul style="list-style-type: none"> isotopic tracers: 30% - 110%; and, stable Carriers: 40% - 110% . <p><i>(ICPT BOA Radiochemistry requirements Part 1 Sec. 2.4.1 and 2.4.2)</i></p>		
4.3.5	<p>The standard material used to prepare tracer solutions is traceable to NIST. If a NIST traceable standard reference material cannot be procured, the standard meets the requirements for a "working reference material" in ASTM C1128.</p> <p><i>(ANSI N42.23 4.1)</i></p>		
4.4	Negative Activities		

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4.4.1	Recurrent problems with significant negative results are investigated and the resolution is documented. <i>(ANSI N42.23 5.2.8, 5.2. and, A.5.2.3; ISO 17025 4.9; NELAC 5.5.4 , and 5.5.2, item o)</i>		
4.5	MDA Determination		
4.5.1	The MDA is calculated for each sample and QC sample analyzed, and for each analyte. <i>(ANSI N42.23, A8)</i>		
4.5.2	The sample and background count times are of the same duration for the MDA equation used or the background count time is greater than the sample count time. <i>(ANSI N42.23, 4.1.3.2)</i>		
4.6	Handling of Environmental Level vs. High Level Samples		

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4.6.1	<p>The laboratory has protocols and controls in place to define how low level and high level samples will be identified, segregated, and processed to prevent sample cross-contamination.</p> <p><i>(ANSI N42.23, A.2.6, ISO 17025 5.3.3)</i></p>		
4.7	Performance Evaluation Programs		
4.7.1	<p>The laboratory participates in a DOE-sponsored performance evaluation program such as QAP or MAPEP for each method used for DOE sample analysis.</p> <p><i>(DOE Order 414.1A; DOE Memoranda (Grimm, 1993; Schmitt 1997, Grumbly, 1994))</i></p>		
4.7.2	<p>Results from recent performance evaluation events are readily accessible.</p> <p><i>(NELAC 5.10.1)</i></p>		

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4.7.3	<p>A corrective action process is initiated when performance evaluation sample results fall outside the established control limits.</p> <p><i>(ANSI N42.23, 5.2.1,5.2.9; DOE Order 414.1A, Criterion 3; ISO 17025, 4.10)</i></p>		
4.8	Sample Preparation		
4.8.1	<p>The laboratory uses techniques to assure the homogeneity of samples such as (but not limited to):</p> <ul style="list-style-type: none"> • liquid samples are mixed thoroughly before removing aliquots for analysis; and • grinding, blending or coning of solid samples yields sub-samples that are representative of the original sample. <p><i>(NELAC 5.10.3)</i></p>		
4.8.2	<p>Pipettes and automatic sample dispensers are uniquely identified through an identification protocol.</p> <p><i>(ANSI N42.23, 5.2.8; ISO 17025, 5.8.2)</i></p>		

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4.8.3	Accuracy of all non-Class A pipettes and automatic sample dispensers used for quantitative measurement is verified monthly or whenever degradation of measuring equipment is suspected. <i>(ANSI N42.23, 5.2.8; NELAC 5.9.4.1)</i>		
4.8.4	Thermometers are uniquely identified through an identification protocol. <i>(ANSI N42.23, 5.2.8; ISO 17025, 5.8.2)</i>		
4.8.5	Liquid-in-glass thermometers are calibrated against a NIST traceable standard at least every five years. <i>(NELAC 5.9.4.1)</i>		
4.8.6	Balances are located in an area where the environment has little or no effect on measurement accuracy. <i>(ISO 17025, 5.3.1 and 5.3.2; NELAC 5.7.1b)</i>		
4.8.7	Balances are calibrated and labeled to that effect annually. <i>(NELAC 5.9.4.1)</i>		

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4.8.8	Daily check weighings are performed using NIST traceable weights in the range used for weighings on the balances. Daily checks are documented in controlled logbooks. (NELAC 5.9.4.1)		
4.8.9	Measures are taken to prevent cross contamination of samples during preparation, such as cleaning grinders between samples. (ANSI N42.23 A.2.6; NELAC D.4.8a)		
4.8.10	At a minimum, non-disposable glassware is fully immersed, soaked and cleaned in a suitable decontaminating/cleaning agent, followed by a thorough acid rinsing, and thorough rinsing with reagent water. (NELAC D.4.8a)		
4.9	Batch Quality Control		
4.9.1	Duplicate spikes, splits and spiked field samples as appropriate are analyzed with every analytical batch (or one per 20 samples, whichever is greater) using the analytes stipulated by the analytical method, applicable regulations or contractual requirement.		

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	(ANSI N42.23, 5.3.1.2)		
4.9.2	Quality control samples are processed in the same manner as field samples. (ANSI N42.23, 5.3.1; NELAC 5.10.1, D4)		
4.9.3	The laboratory has made provision for the following as they relate to the different QC levels: <ul style="list-style-type: none"> • analysis of method and reagent blanks; • analysis of duplicates, spiked samples, spiked laboratory blanks, and reference or control standards such as EPA check standards; • criteria used to establish warning and control limits for the above types of QC samples; • documentation and examples of control data and control charts; • frequency of analyzing blanks and other QC samples; • how data from QC samples are reported and reviewed; and, • who reviews and makes decisions relative to QC data. (ANSI N42.23 A.5.2, DOE Order 414.1A Criterion 3c; NELAC 5.5.3.5, 5.5.4)		
4.10	Standards Preparation		

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4.10.1	Standards and reference materials are traceable to EPA or NIST certified standards, including: <ul style="list-style-type: none"> • initial calibration standards; • continuing calibration standards; • spiking standards; • chemical tracers; and, • counting standards. <p>If a NIST traceable standard reference material cannot be procured, the standard meets the requirements for a "working reference material" in ASTM C1128.</p> <p>(ANSI N42.23, 4.1; NELAC D.4.7)</p>		
4.10.2	Standards are assigned a unique identification number traceable to the original standard. <p>(ANSI N42.23, 5.2.6; ISO 17025, 5.8.2)</p>		
4.10.3	Standard materials are inspected upon receipt to ensure they are accompanied by proper certification documents. <p>(ANSI N 42.23. 5.2.3;, ISO 17025, 5.8.3; NELAC D.4.7)</p>		

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4.10.4	Standards are labeled with the date received, date opened and the expiration date. (NELAC 5.10.5)		
4.10.5	Labels for purchased stock mixtures and reagents contain the following information: <ul style="list-style-type: none"> • date received; • date opened; and, • expiration date. (NELAC 5.10.5)		
4.10.6	The following documentation is maintained for working standards and standards are labeled with the following information: <ul style="list-style-type: none"> • standard identification number; • preparer's initials; • preparation date; • final activity • standard expiration date. (NELAC 5.10.5)		
4.10.7	Standards and reference materials are stored separately from		

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	samples and standards are protected in a controlled cabinet. <i>(ANSI N42.23, A.2.6; ISO 17025, 5.8.4)</i>		
4.10.8	Corrections for radioactive decay and/or ingrowth of progeny have been performed for radionuclide standards. <i>(ANSI N42.23, 4.1.3.22)</i>		
4.10.9	Preparation of standards solutions used for a period of time exceeding one year are verified annually, at a minimum, and documented in a log book. <i>(ICPT BOA Radiochemistry Requirements Part 1 Sect. 2.9.2)</i>		

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4.10.10	<p>The following criteria are met for standards preparation:</p> <ul style="list-style-type: none"> at least 3 verification measurements of a standard are used to determine the mean value and standard deviation of the verification results; certificate value (NOT including any uncertainty) lies within the 95% confidence interval determined from the mean and two sigma standard deviation of the three measurements; and, two-sigma value used for the 95% confidence interval does not exceed 10% of the mean value of the three measurements. <p><i>(ICPT BOA Radiochemistry Requirements Part 1 Sec. 2.9.3 thru 2.9.5)</i></p>		
4.11	Detection Limits		
4.11.1	<p>The lowest concentration that can be measured reliably (detection limit) is clearly established.</p> <p><i>(ANSI N42.23, A.7.4 - A.7.6)</i></p>		
4.12	Instrument Calibration (General)		

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4.12.1	Instrument calibrations are conducted within the following general guidelines: <ul style="list-style-type: none"> • frequency consistent with method or contract-specific requirements; • includes all required parameters; • sources are appropriate for expected radionuclides and counting geometries; and, • where practical, activities of major radionuclides in the counting sources are sufficient to provide counting uncertainties of less than 1%. <i>(ANSI N42.23, A.5.2.1)</i>		
4.12.2	Out-of-calibration equipment is tagged or segregated and not used until it has been re-calibrated. <i>(ANSI N42.23, 5.2.8; DOE Order 414.1A – Criterion 3; ISO 17025, 5.5.7)</i>		
4.12.3	Equipment consistently found to be out of calibration is repaired or replaced. <i>(ANSI N42.23, 5.2.8; DOE Order 414.1A – Criterion 3; ISO 17025, 5.5.7)</i>		

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4.13	Instrument Operations (General)		
4.13.1	All maintenance performed on analytical instrumentation is recorded in a logbook. <i>(ANSI N42.23, 5.2.8; ISO 17025, 5.5.5; NELAC 5.8)</i>		
4.13.2	The identification number of the alpha spectrometer, gamma spectrometer, gas flow proportional counter detector and alpha scintillation detector used to count each sample is recorded as a component of the raw instrument data. <i>(ISO 17025, 5.5.4; NELAC 5.12.3.3)</i>		
4.13.3	Background for alpha spectrometer, gas flow proportional counter, and alpha scintillation detector are rechecked after being subjected to high-activity samples. <i>(ISO 17025, 5.5.7)</i>		
4.13.4	Repair, reconfiguration or replacement of an instrument is followed by verification of calibration of the system. If calibration verification parameters are not met, full calibration is performed. <i>(ANSI N42.23 A.5.1; ISO 17025, 5.10.4.3; NELAC 5.9.4.1,</i>		

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4.13.5	The laboratory identifies and documents the instrument manufacturer, model number, configuration, settings, detector identifications and any repairs or modifications in the instrument maintenance log. <i>(ISO 17025, 5.5.5; NELAC 5.8)</i>		
4.14	Alpha Spectroscopy		
4.14.1	The calibration of each alpha spectrometry detector used to produce data for DOE includes: <ul style="list-style-type: none"> • channel vs. energy calibration - performed at least monthly; • efficiency determination - when the check source count is outside of the acceptable limits of the control chart; and, • background determinations for each ROI - at least monthly. <i>(DOE Order 414.1A Criterion 3d, NELAC D.4.4))</i>		

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4.14.2	Energy Calibration meets the following criteria: <ul style="list-style-type: none"> a curve is fit for Energy (Y-axis) versus Channel (X-axis) and the equation with the slope and Y-intercept for the fit is documented; slope of the equation is ≤ 15 keV/channel; performed using at least three isotopes with the energy range of 3 to 6 MeV; and, final peak energy positions of all observed isotopes are within ± 40 keV of the expected peak energy. <i>(ICPT BOA Radiochemistry Requirements Part 2 Sec. 2.2.4)</i>		
4.14.3	The Background total counts (or counts per unit time) for each target analyte and tracer isotope Region of Interest are analyzed on each detector and documented. <i>(ANSI N42.23, A5.2.3; NELAC D.4.4)</i>		
4.14.4	The analyst monitors tailing and peak overlapping. <i>(ICPT BOA Radiochemistry Requirements Part 2 Sec. 2.1)</i>		

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4.14.5	<p>Efficiency determinations meet the following criteria:</p> <ul style="list-style-type: none"> • efficiency counts for the ROIs are background corrected using the same ROIs for the background unless the background is less than 0.5% of the total counts in the ROI; • efficiency is determined on at least 10,000 net counts in the ROI (after background correction); • check source counts to verify efficiency are determined on at least 2,000 counts; • efficiency error is documented; and, • efficiency check is determined by the check source count and its associated error and limits of acceptability for the check source result are documented. <p><i>(ICPT BOA Radiochemistry Requirements Part 2 Sec. 2.2.6)</i></p>		
4.14.6	<p>Resolution of sample spectra is monitored to assure FWHM of peaks is consistently ≤ 100 keV for all detectors.</p> <p><i>(ICPT BOA Radiochemistry Requirements Part 2 Sec. 2.3.2)</i></p>		
4.14.7	<p>Internal tracer method is used for isotopic specific analysis.</p> <p><i>(ICPT BOA Radiochemistry Requirements Part 2 Sec. 1.1.1)</i></p>		

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4.14.8	Alpha spectrum regions of interest are selected with consistency from analyte to analyte. <i>(ICPT SOW Radiochemistry Requirements Part 2 Sec. 2.3)</i>		
4.14.9	Calibration for energy, background, and efficiency determinations is performed when a new detector is put into service or if repair is performed on an existing detector. <i>(DOE Order 414. 1A, Criterion 3d; ISO 17025,5.10.4.3)</i>		
4.14.10	Tracers have been tested by the laboratory for contribution in the ROIs of the analytes of interest. <i>(ICPT BOA Radiochemistry Requirements Part 2 Sec. 1.1.4)</i>		
4.14.11	The laboratory has established limits for background levels. <i>(ANSI N42.23, A.5.2.3)</i>		
4.14.12	Alpha spectrometry energy calibrations, efficiency calibrations and verifications are performed at a frequency determined by contract or site-specific requirements. <i>(ICPT BOA Radiochemistry Requirements Part 2 Sec. 2.2)</i>		

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4.15	Gas Flow Proportional Counting		
4.15.1	The alpha/beta discrimination method employed is Detector Voltage Adjustment (DVA), Pulse Height Discrimination (PHD) or Rise Time Discrimination. <i>(ANSI N42.25, 4.2)</i>		
4.15.2	Plateau Measurement: <ul style="list-style-type: none"> • voltage range used to determine the plateau is 300 V-500 V; • voltage increase per step is <50 V per step; • slope of the alpha plateau over a range of 100 V is <5% over 100 V for point sources; and, • slope of the beta plateau over a range of 100 V is <5% over 100 V for point sources. <i>(ANSI 42.25, 4.2)</i>		

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4.15.3	For alpha and beta plateau measurements, <ul style="list-style-type: none"> a massless point source is used for α and for β measurement; point source consists of a pure radionuclide; and, active area of the check source is as small as possible (few millimeters in diameter). <i>(ANSI N42.25, 5.2)</i>		
4.15.4	Efficiency calibration sources are: <ul style="list-style-type: none"> type I (NIST Traceable) or Type II (prepared from solutions traceable to NIST); and, type I sources are calibrated using 2 π emission rate by NIST certifying organization. <i>(ANSI N42.25, 5.1)</i>		
4.15.5	Each alpha and beta efficiency calibration standard is made of a pure radionuclide. <i>(ANSI N42.25, 4.5)</i>		
4.15.6	Self-absorption and cross-talk curves consist of at least 7 points, well distributed throughout the mass range.		

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	Self-absorption curves exist for both alpha and beta counting. (ICPT BOA Radiochemistry requirements Part 4 Sect. 2.2.4.1)		
4.15.7	The sources used for the determination of self-absorption and cross talk are of similar isotope content to that of the analytical samples. (ANSI N42.25 6.5,6.6)		
4.15.8	Cross-talk Determination: <ul style="list-style-type: none"> • Am-241 or Po-210 is used to determine the alpha cross-talk; (ANSI N42.25, 6.4) <ul style="list-style-type: none"> • Sr-90/4-90 is used to determine the beta cross-talk; • Vendor soft ware used to calculate results provides for cross-talk correction; and, • Calculations of sample activity performed manually or programmed by the laboratory provide for cross-talk correction. 		
4.15.9	For alpha-only operation, alpha efficiency is determined. For alpha/beta operation, alpha and beta efficiencies are determined simultaneously. (ANSI N42.25, 4.5)		

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4.15.10	The geometry of the calibration sources used for efficiency and self-absorption/cross-talk curves is the same as that of the prepared sample and QC sample planchets. <i>(ANSI N42.25, 4.5)</i>		
4.15.11	Each alpha and beta calibration standard is counted to an accumulation of 10,000 counts. <i>(ICPT BOA Radiochemistry requirements Part 4 Sect. 2.2.4.5)</i>		
4.15.12	Efficiency calibration is performed when one or more of the following occur: <ul style="list-style-type: none"> • a hardware component has been replaced or repaired; • changes have been made to the system (sample preparation procedure changed or gas flow adjusted); or • quality or manufacturer of counting gas has changed. <i>(ANSI N42.25, 4.5)</i>		

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4.15.13	<p>The alpha and beta calibration of each detector used to count analytical samples or QC samples is checked daily except when used for extended count times.</p> <p><i>(ANSI N42.25, 4.2; NELAC D.4.4)</i></p>		
4.15.14	<p>Efficiency check sources are counted to an accumulation of >50,000 counts to verify calibration and measured detector efficiency.</p> <p><i>(ANSI N42.25, 6.6)</i></p>		
4.15.15	<p>Following gas bottle changes, check sources and backgrounds are counted and fall within established limits before samples are counted.</p> <p><i>(ANSI N42.25, 4.1)</i></p>		
4.15.16	<p>Daily background and efficiency check data is documented, retained, and monitored using control charts or tolerance charts.</p> <p><i>(ANSI N42.23, A.5.2, NELAC D.4.4)</i></p>		

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4.15.17	Daily background counts are acquired for a period of time comparable to the samples. <i>(ANSI N42.25, 4.4)</i>		
4.15.18	Background counts are: <ul style="list-style-type: none"> performed quarterly; acquired such that good counting statistics (e.g., for a period of >10,000 seconds) are obtained; counted at the same time of day as sample measurements; and, monitored using control charts or tolerance charts. <i>(ANSI N42.25, 4.4, NELAC D.4.4)</i>		
4.15.19	Samples prepared for counting exhibit uniform of deposition on the planchet and a minimum of material on the side of the planchet. <i>(ICPT BOA Radiochemistry Requirements Part 1 Sect. 1.6)</i>		

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4.15.20	Planchets are: <ul style="list-style-type: none"> thoroughly cleaned before use to ensure that there are no interfering residues or contamination; prepared not to exceed sample weights in excess of the calibrated ranges of established self-absorption curves; and, stored in a manner that preserves their integrity until they are counted. <i>(ICPT BOA Radiochemistry requirements Part 4 Sect. 1.1)</i>		
4.15.21	The counting gas meets GFPC manufacturer's specification or is at least laboratory grade. <i>(ANSI 42.25, B.1)</i>		
4.15.22	Gas flow is on for 24 hours before making measurements and, if high voltage has been off, it is applied 15 minutes prior to making measurements. <i>(ANSI 42.25, B.1)</i>		

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4.16	Gamma Spectrometry		
4.16.1	<p>The gamma detector system consists of detectors suitable for measuring the gamma isotopes of interest in the range of ≥ 0.06 to ≤ 2 MeV with regard to attaining RDLs, bias and precision requirements.</p> <p><i>(ICPT BOA Radiochemistry Requirements Part 6 Sect. 1.1.1)</i></p>		
4.16.2	<p>Detectors are calibrated for the specific geometry and matrix considerations used in the sample analysis.</p> <p><i>(ANSI N42.14, 5.2)</i></p>		
4.16.3	<p>Identification of the reference used for the half-life, abundance and peak energy of all nuclides is documented.</p> <p><i>(ANSI N42.14, 8 and 8.8)</i></p>		
4.16.4	<p>Background measurements are performed on at least a monthly basis.</p> <p><i>(NELAC D4.4)</i></p>		

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4.16.5	For large low-activity samples, the background spectrum is obtained with a blank sample in the same geometry and of the same density. (ANSI N42.14, 6.4)		
4.16.6	The background count is accumulated at for least the same counting time as used in counting the samples to be analyzed. (ANSI N42.14, 6.4)		
4.16.7	A background is collected and documented after any counting chamber changes, i.e. cleaning, liner replacement, or instrument modification. (ISO 10725, 5.10.4.3)		
4.16.8	Samples are counted in the same source-to-detector geometry as used to establish the efficiency, unless the differences can be accurately compensated for by calculation, and the gamma-ray spectrum is accumulated at the same gain as used in the energy calibration. (ANSI N42.14, 6.5)		

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4.16.9	Energy calibration source is counted for a duration to give sufficient peak counts to permit measurement of peak positions with a precision of <0.2 keV. (ANSI N42.14, 5.1)		
4.16.10	Efficiency calibration is performed as a function of energy. (ANSI N42.14, 5.2)		
4.16.11	Software includes corrections for matrix or density attenuation, coincidence summing, radioactive decay, and pulse-pileup. Each correction factor is applied whenever the magnitude is expected to be one-third of the desired accuracy in resulting gamma-ray emission rate. (ANSI N42.14, 6.5)		
4.16.12	Verification of the analysis software has been performed by the vendor using the performance tests outlined in ANSI N42.14, section 8, for the software version in use and the results are documented to the user. (ANSI N42.14, 8)		
4.16.13	The same peak-area measuring technique used to measure the		

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	detector efficiency is used for analysis of samples. <i>(ANSI N42.14, 6.2)</i>		
4.16.14	Sample gamma-ray spectra are accumulated at the same gain used in the energy calibration and for sufficient time to give acceptable statistical uncertainty. <i>(ANSI N42.14, 6.5)</i>		
4.16.15	The energy calibration for each gamma spectrometry system is checked each day of use (for spectrometer with long-term pulse-height stability). <i>(ANSI N42.14, 5.1; NELAC D.4.4)</i>		
4.16.16	Each sample and QC sample spectrum is assessed for acceptability of key peak width and shape, and interference due to superimposed peaks or other sources. <i>(ANSI N42.14, 6.2)</i>		

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4.16.17	<p>Performance testing for Germanium spectrometers is conducted as follows:</p> <ul style="list-style-type: none"> • elapsed time clock is checked at the time the multi-channel analyzer is installed and after any maintenance to the MCA; • DC offset and amplifier pole zero are checked at least annually and after any related maintenance to the spectrometer; • energy calibration checked (daily to semiweekly) with source whose energies are known and cover ROI; • full-energy peak efficiencies are checked (daily to weekly) using a source with long half-life emitting a low energy and high energy gamma-ray; • the same counting geometry is used for efficiency checks with reproducible source-detector distance; • at least 20,000 counts are accumulated for the net peak areas for the peaks used; • deviations in peak counting rates for either the low or high energy peaks outside 2 standard deviations of the mean are investigated; and, • energy resolution at both low and high energies are measured and recorded (during efficiency calibration tests) and unexpected peak width increases are investigated. <p>(ANSI N42.14)</p>		

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4.16.18	<p>Performance testing for Sodium Iodide detectors is conducted as follows:</p> <ul style="list-style-type: none"> • system energy calibration is repeated each day with one or more check sources in the energy ROI; • system count rate precision for one long-lived radionuclide is checked on each day of use; • efficiency calibration is checked at least semiannually using standard sources spanning energy ROI; • ambient background is checked at beginning and end of each day and before and after each batch of samples; • system pulse height resolution is determined a time of installation and at least once each day of use; • performance checks are recorded such that deviations from the norm are readily observable; and, • appropriate action (e.g., confirmation, repair, and recalibration as required) is taken when measured values are outside predetermined limits. <p>(ANSI N42.12, 4.3.5)</p>		

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4.16.19	<p>The NaI detector energy calibration is generated using the following criteria:</p> <ul style="list-style-type: none"> • using reproducible counting geometry at a fixed gain; • a spectrum is generated containing full energy peaks that span the gamma-ray energy region of interest; • the slope and intercept of the calibration curve is determined using two gamma-ray energies near the extremes of the energy region of interest; • energy calibration is determined for each amplifier gain or photomultiplier high-voltage setting used; and • the radioactivity standard source gamma-ray emission rate is decay corrected to time at which count rate is measured. <p>(ANSI N42.14, 4.3.2)</p>		
4.16.20	<p>Samples are counted on NaI detectors using the same counting geometry that was used for the efficiency calibration.</p> <p>(ANSI 42.12, 4.3.4)</p>		
4.16.21	<p>If the system does not have internal dead-time compensation, the dead-time correction is determined experimentally.</p> <p>(ANSI 42.12, 6.7)</p>		

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4.16.22	Gamma-ray emission rate is decay corrected to time at which count rate is measured at the mid-point of the counting period. (ANSI N42.14 6.6.1.1; ANSI N42.12, 6.8)		
4.17	Liquid Scintillation Counting		
4.17.1	Water samples are checked for chemical preservation and results of all checks are recorded. (NELAC 5.11.3)		
4.17.2	For tritium analysis, water samples and the associated QC samples are distilled prior to analysis. The same fraction is collected for samples and the associated QC samples. (ICPT BOA Radiochemistry requirements Part 3 Sect. 1.1.1)		
4.17.3	Sample counting vials are low potassium borosilicate glass or high density polyethylene vials or certified equivalent. (ICPT BOA Radiochemistry requirements Part 3 Sect. 1.2.1)		

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4.17.4	Vials are prepared according to manufacturer's specification for the cocktail. <i>(ICPT BOA Radiochemistry requirements Part 3 Sect. 1.2.2)</i>		
4.17.5	Vials are "dark adapted" for a minimum of 30 minutes or according to the cocktail manufacturer's specifications before counting. <i>(ICPT BOA Radiochemistry Requirements part 3 Sect. 1.2.2)</i>		
4.17.6	Vials are treated to eliminate static prior to counting. <i>(ANSI N42.15, 8.8.3)</i>		
4.17.7	Instrument background vials for all tritium matrices are prepared with low tritium or "dead" water. <ul style="list-style-type: none"> • type of water used to prepare the instrument background vials is explicitly noted on the preparation and counting documentation; • water / cocktail ratio is the same as for the samples; and, • instrument background is determined weekly or with each sample batch. <i>(NELAC D.4.4)</i>		

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4.17.8	Instrument Performance Assessment check sources, background, and Chi-squared test or other test to determine reproducibility of the measured sample count rate are performed after instrument installation, service, or replacement of sealed check sources. (ANSI N42.15, 7.2.2)		
4.17.9	For analysis methods using quench curves to determine individual sample counting efficiency, the quench curve is generated at least yearly and verified after any instrument maintenance. (ICPT BOA Radiochemistry Requirements Part 3 Sec. 2.4.2)		
4.17.10	If the calibration method is constant quench, the efficiency standards are counted weekly or with each counting batch. (ICPT BOA Radiochemistry Requirements Part 3 Sec. 2.4.2)		
4.17.11	For the constant quench method. 10 x E5 counts are accumulated for the efficiency standard. (ANSI N42.15, 7.2.2.1)		

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4.17.12	<p>The SOP for liquid scintillation methods contain the following :</p> <ul style="list-style-type: none"> if the constant quench method of calibration is used, the quench of the sample is within $\pm 5\%$ of the quench efficiency standard. <p><i>(ICPT BOA Radiochemistry Requirements Part 3 Sec. 3.1)</i></p> <ul style="list-style-type: none"> corrective action procedure for sample quench not meeting requirements. <p><i>(ANSI N42.23 5.2.4,5.2.9; DOE Order 414.1A Criterion 3; NELAC 5.10.1.2)</i></p>		
4.17.13	<p>Each sample and QC sample spectrum is assessed for correctly chosen ROIs, acceptability of peak shape, and interferences due to non-target analytes or luminescence.</p> <p><i>(ICPT BOA Radiochemistry Requirements Part 3 Sec. 3.2.2)</i></p>		
4.17.14	<p>Instrument performance is checked each day of use.</p> <p><i>(ANSI N42.15, 7.2.3.1)</i></p>		

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4.17.15	Control charts are maintained for the Instrument Performance Assessment check sources (H-3, C-14, and background). (ANSI N42.15, 7.2.3.1, DOE Order 414.1A Criterion 3c)		
4.17.16	Control chart limits are set at 2 standard deviations. (ANSI N42.15, 7.2.3.4)		
4.17.17	The IPA check sources are counted to accumulate 20,000 counts, or for at least 30 seconds. (ANSI N42.15, 7.2.3.2)		
4.17.18	The flame sealed background check source is counted twice daily for background measurements. (ANSI N42.15, 7.2.3.3)		
4.17.19	The counting time per measurement for the flame sealed background check source is constant to achieve statistically comparable counts. (ANSI N42.15, 7.2.3.3)		

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4.17.20	Each instrument has its own assigned set of check sources that includes a background source. (ANSI N42.15, 8.5)		
4.17.21	The IPA flame-sealed check sources and background source are: <ul style="list-style-type: none"> clearly labeled with the date they were sealed; within 5 years of their seal date; and, are not stored in direct sunlight or under fluorescent lights. (ANSI N42.15, 6.2)		
4.17.22	Check source vials (background, H-3, and C-14 contain 15 mL toluene and 5 g PPO/L (ANSI N42.15,6.1)		
4.17.23	The H-3 check source activity is between 1E+5 to 3E+5 dpm (2 – 5 kBq). (ANSI N42.15, 6.1.1)		

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4.18.24	The C-14 check source activity is between 3E+4 to 1.5 E+5 dpm (0.5 – 3 kBq). <i>(ANSI N42.15, 6.1.1)</i>		
4.19	Kinetic Phosphorescence Analysis		
4.19.1	Water samples are at least evaporated to dryness and wet-ashed. <i>(ASTM D 5174 – 97, 5.1)</i>		
4.19.2	For all low-level uranium analysis, prior to initial use, all new glassware with the exception of cuvettes, are soaked in hot 8 molar nitric acid for at least two hours and then in room temperature 8 molar nitric acid overnight. <i>(NELAC D.4.1.8)</i>		
4.19.3	Reagent water (ASTM Type II or equivalent) is used. <i>(NELAC D.1.6)</i>		

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4.19.4	<p>The SOP specifies the following acceptance criteria:</p> <ul style="list-style-type: none"> phosphorescence lifetimes fall in the range of 150s to 350s; linear regression coefficient of the decay play is greater than 0.96 for samples where the measured concentration is greater than the RDL.; standard addition recovery is greater than 90% when Analyte concentration is in the range of the calibration curve used; reference Ratio is between 0.9 and 1.1; and, continuing calibration check standard is within 10% of the known value; <p><i>(ICPT BOA Radiochemistry Requirements Part 5 Sec. 2.2)</i></p>		
4.19.5	<p>The KPA is calibrated daily when in use.</p> <p><i>(ASTM D 5174 - 97, 9.2)</i></p>		
4.19.6	<p>At least three standards are used for each calibration range. The calibration range includes the range of the samples to be measured.</p>		

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	<i>(ICPT BOA Radiochemistry Requirements Part 5 Sec. 2.3.2)</i>		
4.19.7	<p>The LCS is measured in the same calibration range as the samples in a batch. If measurements are performed in more than one calibration range, then a separate LCS is prepared for each range.</p> <p><i>(ICPT BOA Radiochemistry Requirements Part 5 Sec 2.3.3)</i></p>		
4.19.8	<p>The performance check is prepared separately and at a different concentration from the calibration standards.</p> <p><i>(ASTM D 1574 – 97, 9.2)</i></p>		
4.19.9	<p>The relative bias of the calibration check standard is in the range of -0.10 to +0.10.</p> <p><i>(ICPT BOA Radiochemistry Requirements Part 5 Sect. 2.4)</i></p>		
4.19.10	<p>The Order for performing Instrument Calibration is</p> <ul style="list-style-type: none"> • Background; • Calibration Curve; and, • Calibration Check standard. <p><i>(ICPT BOA Radiochemistry Requirements Part 5 Sec. 2.5)</i></p>		

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4.19.11	Calibration Requirements: <ul style="list-style-type: none"> linear regression coefficient for the calibration curve is ≥ 0.99; Calibration Check Standard is within 10% of the known value. <i>(ICPT BOA Radiochemistry Requirements Part 5 Sect. 2.6)</i>		

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References cited in this checklist:

ANSI N42.12-1994, American National Standard Calibration and Usage of Thallium-Activated Sodium Iodide Detector Systems for Assay of Radionuclides.

ANSI N42.14-1999, American National Standard for Calibration and Use of Germanium Spectrometers for the Measurement of Gamma-Ray Emission Rates of Radionuclides.

ANSI N42.15, American National Check Sources for and Verification of Liquid-Scintillation Counting Systems.

ANSI N42.23-1996, American National Standard Measurement and Associated Instrumentation Quality Assurance for Radioassay Laboratories.

ANSI N42.25-1997, American National Standard Calibration and Usage of Alpha/Beta Proportional Counters.

ASTM D 5174 – 97, American Society for Testing and Materials, Standard Test Method for Trace Uranium in Water by Pulsed-Laser Phosphorimetry.

International Standard ISO/IEC 17025:1999(E), General requirements for the competence of testing and calibration laboratories.

U. S. Department of Energy, 1999, DOE Order 414.1A, Quality Assurance.

National Environmental Laboratory Accreditation Conference, 2000 NELAC Standards, Chapter 5, Quality Systems.

Lines of Inquiry pertaining to requirements in the ICPT BOA may carry reference to NELAC Chapter 5 if the document carried information pertaining to the line of inquiry.

NELAC encompasses the necessary scientific testing to serve the needs of the US Environmental Protection Agency (EPA), and other federal agencies involved in the generation and use of environmental data, where such generation or use is mandated by EPA statutes and pursuant regulations.

ICPT Basic Ordering Agreement

Sole reference to ICPT Basic Ordering Agreement (BOA) indicates a specific requirement for data produced under the ICPT BOA. For applications of this checklist not aligned with the ICPT BOA, this would be considered a Department of Energy (DOE) best practice.

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